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Experimental validation of a versatile test bench for thermal contraction measurements down to 1.8 K

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Abstract. The use of cryogenics plays a major role in the operation of modern high-energy accelerators and ensures, for instance, the superconducting state of beam-guiding and focusing magnets. A profound understanding of the thermo-mechanical behavior of the structures and components is therefore crucial, particularly for the strain-sensitive Nb_3Sn-based superconducting coils. Inaccuracies in considering processes like thermal contraction can substantially impact the performance of the magnets. However, the scarcity of commercial thermal contraction measurement devices capable of examining these materials down to temperatures as low as 2 K has limited our knowledge of the thermal contraction within these complex systems. The Mechanical Measurement Laboratory at CERN has thus initiated the development of a customized dilatometric test bench in collaboration with the external company attoCUBE. The resulting setup features an optical displacement sensor based on Fiber-Optic Fabry-Pérot interferometry and, with its integration into a closed-cycle cryostat, this setup accommodates temperatures from room temperature down to a minimum of 1.8 K. Preliminary tests on known materials indicated the presence of a systematic error which was corrected by a calibration study using certified single-crystal silicon from the National Metrology Institute of Japan (NMIJ) as reference material. This study served simultaneously as the base for an uncertainty analysis to determine the remaining measurement uncertainty. Using the methods of the *Guide to the expression of uncertainty in measurement* (GUM), an uncertainty better than 0.01×10^{-3} in $\Delta L/L_0$ over the entire temperature range was obtained. Given this level of uncertainty, this setup is well-balanced between accuracy and time efficiency, facilitating dynamic measurements across the entire low-temperature range within a 10-hour timeframe for a single measurement.

1 Introduction

Cryogenics are increasingly relevant to our daily life and are encountered in energy [1] and food industry [2], as well as medicine [3] and many science applications. For high-energy accelerators like the LHC at CERN, the use of cryogenics is indispensable to the employment of superconductors in beam-guiding and focusing magnets, accelerating cavities, and various detector modules. It is vital to understand their thermo-mechanical behavior as these components undergo a significant temperature change from installation to operation, and are generally integrated by a multitude of materials. The performance of Nb_3Sn-based superconducting coils, for instance, is substantially affected by strain due to the extreme brittleness of the Nb_3Sn [4].

A major contributor to the thermo-mechanical behavior of an object is thermal contraction, which quantifies the dimensional change of solids during a cooling process. Hence, it represents indispensable information for the thermo-mechanical design of structures in cryogenic environments. While thermal

contraction data of common materials is available in the existing literature [5, 6], it is often necessary to perform experimental studies for less used and novel materials, or in case the literature values do not satisfy the demanded accuracy. Yet, commercial systems capable of such measurements, as in [7], are presently still scarce and may imply various constraints, such as limitations in the sample size or the examinable materials.
Given the ubiquitous presence of cryogenic environments at CERN and the demand for experimental data on the thermal contraction of many advanced materials and composites, the Mechanical Measurement Laboratory initiated a collaboration with the external company attoCUBE to develop mutually a thermal contraction test bench. This setup, based on an off-the-shelf closed-cycle cryostats and an interferometric displacement sensor, aims to balance accuracy and sample throughput while avoiding any essential material limitations.
This paper describes the experimental setup and discusses its characteristics and calibration. Furthermore, it gives an overview of the estimation of the measurement uncertainty using single-crystal silicon as a certified reference material.

2 Experimental setup

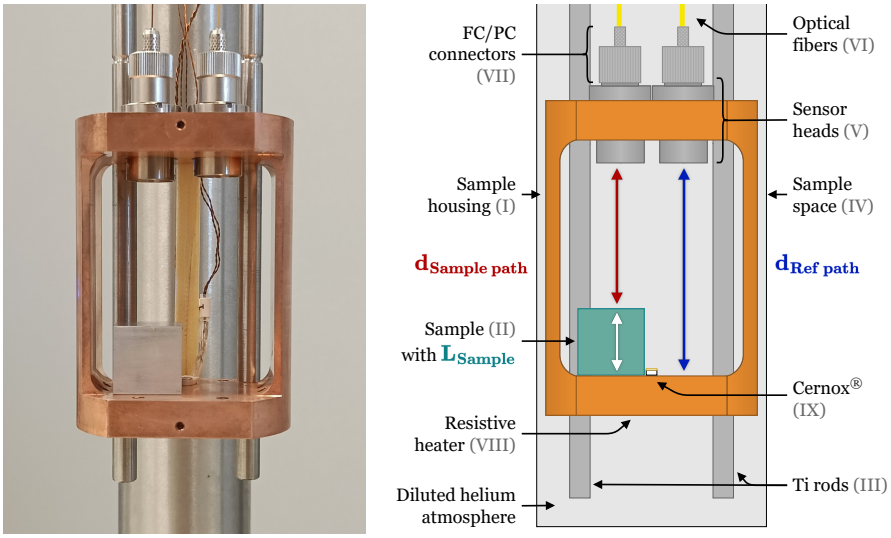
2.1 Cryogenic environment

The dilatometric test bench is a customized insert for the attoDRY2100 from attoCUBE, which is a closed-cycle cryostat capable of varying the temperature in its sample space from 300 K to 1.8 K. The cooling power is provided by two cooling cycles based on a pulse-tube cryocooler, and a secondary cycle using the Joule-Thomson effect. This combination realizes a cool-down to 1.8 K within 3 h to 4 h. An integrated resistive heater counters this cooling and provides temperature control in dynamic and steady-state conditions.
A distinctive characteristic of the system is the low-vibration sample space, despite the presence of a pulse-tube cryocooler which inherently induces vibrations. This is achieved by exchanging heat within the sample space solely via gas conduction and convection using a helium atmosphere. Hence, the sample platform is suspended via G10 rods from the top of the cryostat and is not in any direct contact with the cold walls of the sample space. However, this entails the need for a certain minimum amount of helium gas in the sample space to facilitate convection between the sample platform and the walls of the sample space. Consequently, the operation in a vacuum is not applicable unless other means of heat transfer are implemented.

2.2 Dilatometric test bench

Fiber-Optic Fabry-Pérot interferometry (FFPI) forms the basis for the dilatometric test bench. It thus takes advantage of the resilience of FFPI to harsh environments, including strong magnetic fields and cryogenic temperatures, which is further supported by the possible compactness of technical implementations of FFPI. [8]
Figure 1 illustrates the dilatometric setup. Its structure is given by the sample housing (I), a copper uni-body equipped with optical and thermal components. The sample (II) is situated inside the cavity of the sample housing, whose dimensions are presently designed for samples with 15 mm height. G10 and titanium rods (III) fixate the suspended sample housing at the bottom of the insert, placing the setup in the sample space (IV) when inserted into the cryostat.
The optical components, namely the two so-called sensor heads (V), are threaded at the top of the copper body, each containing a collimating lens with a different focal length. According to the placement of the sample, the sensor heads are distinguished into a reference side and a sample side. Optical fibers (VI) are attached at the top of both sensor heads using FC/PC connectors (VII). The fibers connect both sensor heads via feed-throughs to the interferometric displacement sensor IDS3010 from attoCUBE, which is located outside of the cryostat. Based on this arrangement, the IDS3010 can detect changes in the distance between the sensor heads and the target surface with nanometer accuracy, provided that the target surface is at the correct distance and exhibits adequate reflectivity. More information on the working principle of FFPI and the IDS3010 is given by Thurner [8, 9].
The temperature control is realized by a resistive heater (VIII) and a Cernox® sensor (IX). While the Cernox is placed just next to the sample to minimize the temperature error between the measured and actual sample temperature, the heater is installed in the bottom of the housing. This heater is however only supplementary to the temperature control to increase the reactivity of the controller.

The measurement principle is indicated in Figure 1b and follows a differential approach. As mentioned before, the IDS3010 monitors continuously the changes of the two distances $d_{Sample\ path}$ and $d_{Ref\ path}$



(a) (b)

Figure 1: The dilatometric test bench consists of a copper cavity with two sensor heads, allowing to detect the displacement along the two optical paths using FFPI. The dilatation of solely the sample can be determined by subtracting the displacements along the two paths.

according to their beam path. Given the structural equivalent of $d_{Ref\ path}$, the reference sensor head detects any dilatation of the sample housing. In parallel, the displacements along the sample path result from a simple combination of length changes of the sample housing $\Delta L_{Sample\ housing}$ and the sample ΔL_{Sample}:

$$\Delta d_{Sample\ path} = \Delta L_{Sample\ housing} - \Delta L_{Sample} \tag{1}$$

Accordingly, the contraction of the sample can be extracted from Equation (1) by subtracting these two displacements, as indicated by Equation (2). Here, the dilatation is expressed as the relative change of length $\Delta L/L_0$, which is relative to the sample length $L_{0,\ Sample}$ at the reference temperature $T_{Ref} = 293\,\mathrm{K}$. $d_i(T)$ corresponds to the distance of one of the two beam paths at the temperature T. Vertical thermal gradients are not regarded in this equation as they are compensated for by considering the dilatation of both beam paths.

$$\left(\frac{\Delta L}{L_0}(T)\right)_{Sample} = \frac{\big(d(T) - d(T_{Ref})\big)_{Ref\ path} - \big(d(T) - d(T_{Ref})\big)_{Sample\ path}}{L_{0,\ Sample}(T_{Ref})} \tag{2}$$

2.3 Sample preparation

In order to ensure accurate detection of the interference patterns and thus of the displacements, a main requirement of the IDS3010 is an adequate target surface reflectivity at the sensor's wavelength of 1530 nm [10, 8]. Therefore, the sample preparation generally involves optimizing the surface reflectivity. For many materials, such as metals, simple polishing with sandpaper is sufficient, while others, such as various polymers or composites, may require an additional coating step. To maintain this coating procedure reasonably simple, a standard aluminum foil of about 15 µm thickness is bonded on the sample surface using a commercial epoxy glue suitable for cryogenic environments. A polishing with fine sandpaper follows to adjust the reflectivity.

This method yielded generally a coating thickness of 10 µm to 20 µm after polishing, which corresponds to about 0.1 % of the sample thickness. Although the induced error is not negligible, this coating technique represents a fast and simple solution without any additional treatments that could affect the sample material. Thinner coatings should however be considered for measurements with high accuracy or of materials with a relatively low contraction.

2.4 Measurement procedure

A measurement starts in general with the determination of the reference length $L_0(T_{Ref})$ of the sample using a micrometer. Subsequently, the setup including sample are inserted into the cryostat, where the sample space is still at room temperature. After evacuating and refilling the sample space with a low helium pressure, the cool-down to 1.8 K is initiated and takes about 4 h. The actual measurement takes place during the following dynamic heat-up. The dilatation is recorded with the IDS3010 while the heating rate is controlled to $1\,\mathrm{K\,min^{-1}}$ using the two resistive heaters. When the reference temperature $T_{Ref} = 293\,\mathrm{K}$ in the sample space is reached, the controller switches to a steady-state mode. A full measurement cycle takes thus about 10 h.

3 Calibration

During the first tests on well-known materials with the dilatometric setup, a significant but consistent deviation was observed. This systematic deviation was identified to be caused by a relative movement of the sensor heads due to differential thermal contraction. Given that the sample housing is based on copper, the sensor heads, fabricated from titanium grade 5, are only subject to half of the thermal expansion of the sample housing. Thus, it is suspected that the threaded connection within the opto-mechanical chain cannot maintain a common reference plane for both sensor heads. However, as this systematic error is repeatable, it can be eliminated by calibration using a reference material.

3.1 Reference material and test matrix

Single-crystal silicon (SCS) is a common calibration material for thermal expansion measurement instruments due to its high material consistency, low and isotropic expansion, as well as high thermal conductivity [11]. Certified samples are provided by the National Metrology Institute of Japan (NMIJ) [12] with thermal contraction data from room temperature down to 20 K. The uncertainty of the reference data-set is in the range of ppb [12], and thus negligible compared to the test bench discussed in this paper.
Three samples were extracted from a certified reference specimen of SCS received from the NMIJ. Each of the three samples underwent five tests, with each test series comprising two consecutive measurement cycles according to the measurement procedure mentioned beforehand. A summary of the study is given in Table 1.

Table 1: Calibration test matrix using single-crystal silicon as certified reference material [12].

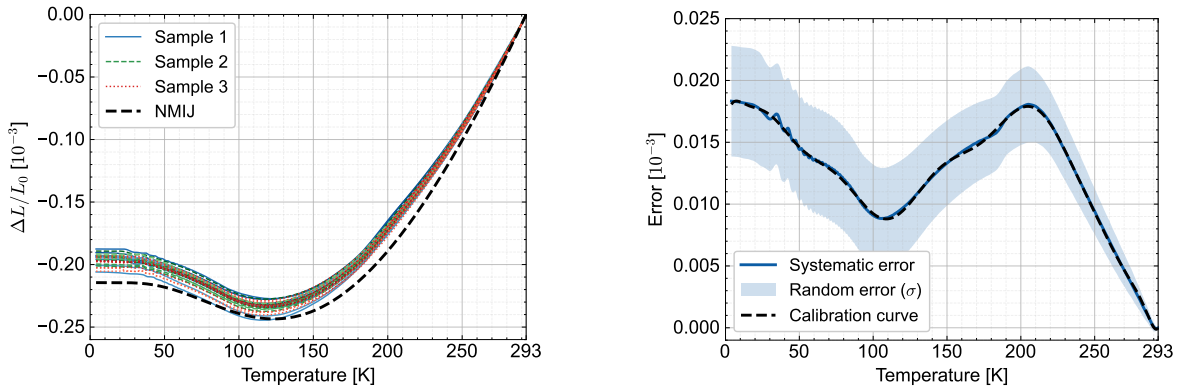
Number of samples	3
Number of tests per sample	5
Number of measurements per test	2
Total number of measurements	30

3.2 Results

The outcomes of all measurements were compared with the reference data provided by the NMIJ. The data-set was however extended to 2 K, assuming that the expansivity α is $0\times10^{-6}\,\mathrm{K^{-1}}$ below 20 K, which is reasoned by the contraction data on SCS given in [11]. The measured relative dilatation is displayed together with the reference data set from the NMIJ over temperature in Figure 2a. The systematic error was subsequently derived by comparing an average of the measured values with the reference data. This is visualized in Figure 2b together with the random error, emphasizing that the systematic difference is four times larger than the dispersion. Lastly, the calibration curve is displayed, which fits the systematic error using a polynomial. The mean error induced by fitting the calibration data is less than $u_{Cal,\ Fit} = 2\times10^{-7}$ and thus negligible.

4 Uncertainty estimation

The uncertainty of the measurements on SCS was analyzed using the *Guide to the expression of uncertainty in measurement* (GUM) [13]. Here, two uncertainties are distinguished, a type A and a type B uncertainty. The type A uncertainty u_A expresses the random dispersion of a measurement and can be described by the variance of an extensive test series [13]. Given the study on SCS for the calibration, u_A was thus determined using the standard deviation of this study with 29 degrees of freedom (see Figure 2b).



(a) Raw data and reference curve

(b) Systematic and random error

Figure 2: (a) Raw data of the measurements on single-crystal silicon next to the reference curve of the NMIJ. (b) The calibration curve is deduced from the systematic error between the mean of all measurements and the reference data. The random error indicates the dispersion among the measurements.

The type B evaluation of uncertainty u_B is obtained from information other than statistical means [13]. In the dilatometric setup, this comprises, for instance, the uncertainty in the reference length determination (L_0), the calibration, the displacement monitoring (ΔL), and the temperature sensing (T). The latter two sources of error are however the most significant ones.
attoCUBE specifies the uncertainty for displacement measurements with the IDS3010 as 0.4 ppm times the target distance [10]. Whilst this estimate neglects any effect on this uncertainty by environmental changes, such as a varying refractive index, it is assumed that their impact is compensated for by the calibration and the type A uncertainty.
The uncertainty induced by the temperature measurement $u_{B,T}$ results from a multiplication of the expansivity of the material and the uncertainty in the measured temperature. The estimate of the uncertainty in the temperature sensing comprises the uncertainty of the Cernox sensor, as well as the error due to the positioning and mounting of the sensor. The latter is estimated based on tests of the temperature distribution over the dilatometric test bench.
An overview of the outcomes of the GUM analysis is visualized in Figure 3. It illustrates the relevant individual uncertainties u_i in the relative change of length $\Delta L/L_0$, and the combined and expanded uncertainty. The expanded uncertainty results from the combined uncertainty by applying the coverage factor $k = 2$, in order to elevate the confidence level to about 95 %. The uncertainty due to the calibration is not displayed due to its insignificance compared to the other contributors.

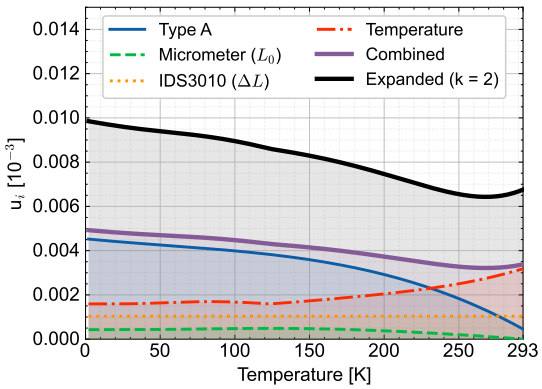


Figure 3: Based on the GUM, the main uncertainty drivers were estimated and are shown together with the combined and expanded uncertainty ($k = 2$).

The graph highlights that the measurements on SCS were performed with an uncertainty of better than 0.01×10^{-3} in the relative change of length over the entire temperature range. Considering the material dependence of this measurement uncertainty, this allows to determine the thermal contraction of common engineering materials like copper or aluminum with an uncertainty of better than 1 %.

5 Conclusion

A dilatometric test bench using a Fiber-Optic Fabry-Pérot interferometer to measure thermal contraction from room temperature down to 1.8 K has been developed and validated. A calibration curve has been determined using certified single-crystal silicon as a reference material. The uncertainty of the test bench has been analyzed based on the methods of the *Guide to the expression of uncertainty in measurement*, which yielded an expanded uncertainty of better than 0.01×10^{-3} for the calibration test series on single-crystal silicon. Considering this level of uncertainty, the setup effectively balances accuracy and time efficiency, as an entire dynamic measurement cycle is conducted in less than 10 h. In addition, by using a simple coating method, the setup is free of limitations on the materials to be examined.
Presently, the test bench requires calibration due to differential thermal expansivity at the interface between the sample housing and sensor heads. A solution is currently in progress, allowing to avoid the calibration and possibly improve the uncertainty further by reducing the dispersion in the measurements.

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